

analysis

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THE SPECIALIST AND JACK

Those of us from small towns or simpler times can remember the "Jack-of-all-Trades". He was considered the best informed, most independent, and most self-sufficient man around. Never out of work and always in demand.

The developing environmental analytical laboratory of the 1950's and 60's guaranteed a key role for a technically-oriented Jack. He knew the chemistry behind any sample, he knew his separation tables, and armed with the arcane tools of his trade like the Gooch crucible, the policeman, the bunsen burner, he could identify many of the fundamental problems in the environment.

In the 1970's the perception of environmental damage and the tools needed to detect it began to change. The bunsen burner and flame testing yielded to laminar flow burners encased in flame photometers. The chain balance, damper balance, and endless combinations of unequal-arm fulcrum balances went the way of the pterodactyl, to be replaced by bland, inscrutable little boxes with one or two knobs on them. Sleek new analytical systems evolved, like atomic absorption spectrophotometers, gas chromatographs and emission and mass spectrometers. These systems were automated. Automated systems were computerized.

Automated, computerized equipment systems do more than just crank out more analyses; they also force an unprecedented degree of specialization onto each technician and scientist. There are numerous instances of analysts devoting all available time to the determination of one parameter (or family of parameters), on one matrix, using one particular instrument system. A man such as Jack has nearly become an anachronism.

Or has he? The 1980's appear to be bringing forth a new set of problems that cannot be solved by churning out more automated data, regardless of the degree of analytical sophistication.

Acid rain provides an example of a problem requiring a more generalistic approach. The researcher of this phenomenon must sift data on pH in water, rain and snow; he must relate this to lake alkalinity; he must be able to devise a means of examining the effects of water and dilute acid on soil, as well as the subtle interactions between soil and vegetation under varying pH conditions. The challenge to the analyst is tremendous. Custom-made analytical procedures need to be devised, means of speciating various cations (such as "free" aluminum) and anions require development, and a more catholic approach to analysis will be demanded by the need to measure miniscule but fundamental changes in the environment.

Jack may be saved, not by the chemists, but by the geologists, biologists, and botanists.

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A CONCENTRATION TECHNIQUE FOR NON-VOLATILE MUTAGENS IN ENVIRONMENTAL SAMPLES

A simple and efficient method has been developed for the concentration of non-volatile organic compounds with boiling points greater than 200°C from aqueous environmental samples. This technique was developed specifically to prepare concentrates for bacterial mutagenicity assays. It was designed to complement the existing concentration technique for volatile organics with boiling points greater than 60°C and less than 200°C.

Tap water samples were spiked with the desired compounds dissolved in DMSO, representing a wide spectrum of polarity, volatility, and potential for mutagenicity/carcinogenicity. Some of these standards included: propylene glycol, 2,4,5-trichlorophenol, 1-naphthylamine, lindane, anthracene and dioctylphthalate. After solvent extraction with methylene chloride, the aqueous phase was concentrated by rotary evaporation under vacuum. Recoveries of the standards were determined by gas chromatography for the methylene chloride extract and the rotary evaporator concentrate. These concentrates were combined, then distilled at a vacuum of 0.1 Torr or less to reduce the volume of DMSO to the desired amount.

The method was evaluated by varying several parameters: sample volume, spike concentration and number of compounds spiked at one time. The sample volumes tested were 1, 2, 4 and 8 litres, at a spike concentration of 2 mg/L. At a constant volume of 1 litre, the spike concentration was varied from 250 µg/L to 10 mg/L. Standards were evaluated individually or in mixtures of up to seven compounds.

As inorganic salts were anticipated to be present in the types of environmental samples treated and could potentially interfere with mutagenicity testing, a centrifugation step was incorporated into the method, for removal of such compounds from the DMSO concentrate. The amount of inorganic salts removed by this procedure was determined.

Another potential interference with mutagenicity testing was the presence of bacterial contamination in the final concentrate. Studies were undertaken to ensure that the DMSO concentrate was free of bacteria and remained so after storage of up to four weeks.

This concentration technique has been successfully applied to environmental samples, and is being integrated into the mutagenicity testing program for industrial effluents in Ontario.

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VEGETATION DAMAGE CAUSED BY TAR FUMES

While claims of vegetation damage by fumes from street and roof-tarring operations have repeatedly been received by the Ministry, it has until now been difficult to prove such allegations analytically with any degree of certainty. Reasons for these difficulties include the irreversible change and, consequently, loss of phytotoxic compounds contained in tar vapours by reaction with the plant tissue. Also volatility of potentially phytotoxic components and/or insufficient sensitivity of analytical methods may similarly prevent detection and identification of minute amounts of such material on affected plant surface areas.

At the request of the Phytotoxicology Section, ARB, laboratory investigations have now revealed that a suitable method for tracing vegetation damage to tarring operations can be based upon the determination of polynuclear aromatic hydrocarbons (PAH) deposited as fallout on such vegetation. The affected leaves are extracted with hexane, the extract taken to dryness under vacuum and the residue dissolved in acetonitrile. This solution, after suitable dilution, is analysed by high pressure liquid chromatography in conjunction with fluorescence measurements. Analysis of vegetation samples with extensive damage from nearby tarring operations showed PAH compounds present at up to 170 times the amounts found on control samples of the same vegetation species.

While PAH compounds themselves are not considered particularly harmful to vegetation, other components in tar vapours, such as phenols, creosote and so on, can cause extensive plant damage. Thus, the presence of PAH compounds in areas affected by tar fumes can serve as an effective indicator for the source from which damaging emissions have been generated. Table I shows some typical values obtained for PAHs from solvent extracted zucchini leaves.

Table I

| <u>Compound</u> <u>Analysed</u> | <u>Damaged Leaves</u> <u>ng/g</u> | <u>Control Leaves</u> <u>ng/g</u> |
|------------------------------------|--------------------------------------|--------------------------------------|
| 1) Fluoranthene | 1613.6 | 9.36 |
| 2) Perylene | 697.5 | 8.94 |
| 3) Benzo(k)pyrene | 596.5 | 7.63 |
| 4) Benzo(a)pyrene | 303.5 | 2.79 |
| 5) Benzo(g,h,i)perylene | 122.0 | 10.95 |
| 6) o-Phenylenepyrene | 202.4 | 6.42 |

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A REGIONAL LABORATORY

In attempting to write an informative article on environmental analysis, based on the achievements or experiences of a regional laboratory, one is forced to the conclusion that it is virtually impossible to present any evidence of technological innovation produced with the limited resources available. In a large laboratory complex, a much higher degree of specialization exists, to the point where an analyst may devote all his time to a particular analysis. In most cases, these analyses are performed on samples unfamiliar to the analyst, and he may have little appreciation of the significance of the final result. It is imperative, therefore, that he perform rigorous quality control on his analyses to ensure that the test method is operating satisfactorily. Considerable time and effort must also be spent on method evaluation by determining possible interferences, recovery of known concentrations, and reproducibility of duplicate analyses. These studies reassure the analyst that the results which he generates are accurate, and serve to advance the science of analysis.

A regional analyst is more familiar with the source of the sample and often has a shrewd idea of the concentration level for most of the requested analyses. He also has an opportunity to discuss the sample with the person submitting it, and hence, gain an appreciation of the problem which generated the sample. The regional analyst, therefore, can anticipate the results of analysis and assess the degree of accuracy required for the information to be useful to the sampler. Thus it might be said that the comparison between the central laboratory and the regional laboratory is that of a starlet and the harlot. The starlet is displaying a high profile and seeking recognition, while the harlot is trying to hustle a living by providing a service without attracting undue attention.

Articles based on regional laboratory experience are, therefore, most likely to be based on examples of client pleasing rather than demonstrations of technical excellence. A frequent occurrence in the Kingston Laboratory is the appearance of a person who is concerned about "pesticides" in his well. One such person appeared to have some grounds for concern since his well was in close proximity to a large industrial facility.

imity to farmland which was intensively cultivated. It was logical to assume that migration of fertilizer would occur in advance of the less mobile pesticides, so his well water was tested for nitrates to further assess the potential for contamination. Elevated nitrate levels were found, the regional pesticide officer contacted, and a sample was forwarded to the Pesticide Laboratory for appropriate analysis based on information obtained by the Pesticide Officer.

On another occasion, an agitated gentleman appeared in the laboratory with a small chemical sample. His problem, as explained, was that the drinking water in his plant tasted so foul that his workers were prepared to walk out. The sample was found to have a pH of 3.2, with chloride, and (estimated)

sulphate concentrations consistent with the normal analysis for water in the municipality, where the plant was located. With this information, it was suggested that carbon dioxide might be entering his waterline. Investigation at the plant by an abatement officer proved that the problem was the soft drink vending machine which was connected to the water supply without a backflow preventer.

These examples demonstrate one role played by a regional laboratory which is to provide chemical and (microbiological) expertise in support of field activities, and conversely, to obtain field confirmation of analytical results.

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THE DETERMINATION OF FREE CYANIDE BY GAS DIALYSIS

Industrial waste streams vary widely in their content of cyanide compounds. These may be highly toxic simple cyanides and hydrogen cyanide, found in plating and metal recovery operations wastes, as well as less toxic compounds such as iron cyanide and thiocyanate in wastes from coking operations.

The determination of the total cyanide is a relatively simple procedure. The determination of the free cyanide fraction, however, in the presence of interferences such as sulfide and thiocyanate, is more difficult. Some methods determine free cyanide as the difference between results obtained on treated and untreated aliquots. These methods are generally tedious and may depend upon small differences between two large numbers, resulting in a less precise value for free cyanide.

A direct method for free cyanide has been developed, which employs sensitive automated colourimetry and an automated gas dialysis procedure for the isolation of the cyanide from interferences. As an acidified sample containing cyanide flows past one side of the dialysis membrane, the gaseous HCN diffuses through the membrane and is swept away from the other side by a parallel flowing alkaline recipient stream. This solution, free of all non-gaseous products, is introduced to the automated colourimetric system for the analysis of free cyanide.

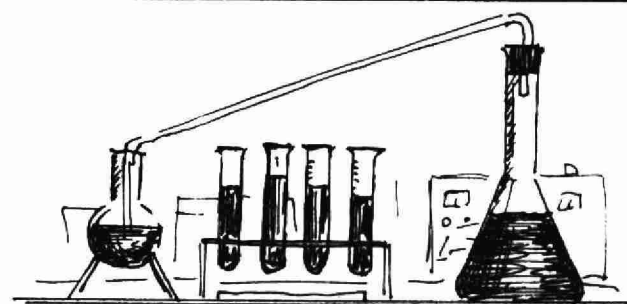
The dialysis membrane consists of Teflon membrane, in the form of a 1" X 4" strip of

Teflon pipe thread tape or a 0.2 μ m Teflon filter material sandwiched between two cellulose-acetate membranes. The Teflon alone was found to be very sensitive and selective (allowing only HCN gas to pass through) but lost selectivity as the flowing solutions "wore out" the membrane. The cellulose acetate is essentially non-selective and serves to protect the Teflon from the flowing solutions without significantly affecting its sensitivity.

This combination of materials has permitted the low level determination of free cyanide in the presence of high level of thiocyanate and sulfides. The system is capable of extended operation without loss of selectivity or sensitivity. It is inexpensive and membrane materials are readily available.

The gas dialysis system responds linearly to increasing cyanide concentrations and under current operation conditions, has a detection limit of 1 ppb for a range of 0 to 400 ppb. Precision of the test is 2 ppb over the range of calibration. Spike recoveries are 100%.

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Daphnia Magna (Strauss)

In 1938 at the University of Toronto, a group of young Canadian physicists constructed the first electron microscope in North America. For its theoretical basis, this Toronto microscope owed almost everything to the pioneer work done by the European scientists in the early 1930's. This achievement was regarded as one of the major engineering successes of the first half of the century. These microscopes soon became widely distributed throughout the world and contributed to developments in ultrastructure research in Physics, Materials Science, Biology and Medicine.

Today, 40 years after the advent of the first commercial electron microscope, researchers are taking advantage of the large number of interactions that occur when a focused electron beam impinges on a specimen surface. Among the characteristic specimen-related signals produced are secondary electrons, backscattered electrons, transmitted electrons, Auger electrons, characteristic and continuum x-rays, cathodoluminescence and electron beam-induced currents.

The term "analytical electron microscopy" (AEM) describes the microcharacterization of any material, using as much as possible of the information produced by such electron-solid interactions. Along with the increasing interest in AEM, advanced techniques for image optimization and fully engineered facilities for a complete range of detection analysis and processing possibilities are now available to users. Fully automated quantita-

tive and qualitative analysis of all elements with atomic number greater than 4, can be obtained using combined energy and wavelength dispersive analysis. On-line image quantitation (size, shape, counts and statistical data) can be derived from scanning electron microscopy (SEM) images and fully automated coordination with x-ray analysis can be obtained.

With the rapid advances in this field, and the numerous advantages these systems offer over more classical techniques, it is important that potential users be informed of the ever-expanding applications of electron microscopy. With this aim in mind, a day-long symposium on electron microscopy and its application is being planned for Wednesday, February 3, 1982, at the Bristol Place Hotel in Toronto. If you would like further information about this symposium please contact:

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AMMONIA DETERMINATION BY GAS PHASE ATOMIC ABSORPTION SPECTROMETRY

Gaseous ammonia absorbs radiation in the ultraviolet region of the spectrum with maxima at 193.4, 197.2, 201.0, 204.8, 208.6 and 212.6 nm. The sensitivity of absorption decreases with increasing wavelength. Ammonia, liberated from a hot solution by means of a strong base, is separated from the liquid phase and passed through a windowless quartz or glass "T" cell aligned in the light path of an atomic absorption spectrophotometer. An arsenic hollow cathode lamp is used as the source of radiation. The monochromator is tuned to 197.2 nm with the spectral band width set at 1 nm. The absorption signals are recorded.

The system has been automated by interfacing a Technicon proportioning pump, sampler and heating bath with an atomic absorption spectrophotometer. Nitrogen is used as a carrier gas. Sensitivity and detection limit of the method are 0.20 and 0.10 ug NH_3/mL , respectively. The calibration curve is linear between 0 and 40 ug NH_3/mL . Samples containing high concentrations of ammonium ion may be determined at less sensitive absorption maxima. The precision of the method is 1.0% relative standard deviation (RSD) at 4.0 ug NH_3/mL .

Table 1

Free Ammonia in Sewage Effluents (ug NH₃/mL)

| <u>GPMA^a</u> <u>Method</u> | | <u>Conway</u> <u>Method</u> |
|--|--------|--------------------------------|
| Raw | Boiled | |
| 34.0 | 32.4 | 35.0 |
| 38.9 | 24.8 | 27.8 |
| 11.6 | 11.0 | 11.8 |
| 8.9 | 8.2 | 9.0 |
| 8.7 | 8.2 | 10.7 |
| 43.0 | 45.6 | 42.0 |
| 10.9 | 10.9 | 10.8 |
| 13.8 | 13.8 | 13.0 |
| 20.0 | 19.7 | 17.7 |

^a gas phase molecular absorption

The method is relatively free from most interferences. Volatile organic compounds interfere but they can be easily removed by boiling the sample solutions before analysis. Up to 140 determinations can be performed

in one man-day. Table 1 shows the agreement between the proposed method and a standard titrimetric method for ammonia.

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A NEW EXTRACTION METHOD FOR THE DETERMINATION OF PCB AND ORGANOCHLORINE PESTICIDES IN BIOLOGICAL TISSUES

The analysis of various types of biological tissues (fish, shellfish, mammals, birds, etc.) is a common requirement for organic residue labs.

The Laboratory's Pesticides Section has compared its existing method of tissue extraction with a modified version of an acid

digestion procedure developed by Dow Chemical (Canada) Ltd. for the analysis of dioxin in fish.

The current method of tissue analysis involves the dissection of the biotic material, followed by grinding with a Virtis homogenizer, maceration and desiccation, using anhydrous sodium sulphate. The dry fish powder is leached in a glass column with 240 mL of dichloromethane. The extract is then concentrated to 100 mL for further treatment.

The new method developed involves the acid digestion of the previously ground tissue using 20 mL concentrated HCl in a 50 mL screw cap culture tube. The acid/tissue digestion is performed on a rotary (30-45 rpm) extraction unit and requires 1½ - 3 hours for total tissue dissolution, dependent on lipid content, extraneous matter, bones, scales, etc. The resulting solution is then extracted twice with 20 mL dichloromethane and the extracts are neutralized, dried and dissolved in 100 mL solvent. The new method is quicker, more thorough and requires less reagents, glassware and manpower than the current method.

Comparative analyses were done on 50 fish tissues and a series of blanks, spikes and duplicates. The compounds analysed included PCB, HCB, Heptachlor, Aldrin, ppDDE, Mirex, alpha BHC, beta BHC, gamma BHC, alpha and gamma Chlordane, opDDT, ppDDD and ppDDT.

The acid digestion method produced very comparable results to that of the column extraction technique. Spikes of all components indicated excellent recoveries.

Both methods were judged sufficient to declare the methods equivalent for the analysis of the compounds tested. Regression analysis and confidence testing (paired T t=0.05) indicated no significant difference between the two methods.

The only compound for which there was a difference between the two methods was alpha Chlordane. This difference is ascribed to other methodological approximations and is not viewed as an indication of a bias in the acid digestion procedure.

Although the acid digestion procedure did not show the expected increase in extraction efficiency, the simplicity of the method, the decrease in solvent usage, as well as the applicability of the method to a wider range of tissue types made it very attractive. Additionally, preliminary results on recoveries of other pesticides and industrial organics including chlorinated benzenes, chlorophenols, triazines and phthalates indicate the applicability of the method to a wide range of commonly analysed contaminants in a single sample extract.

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ENVIRONMENT ONTARIO RESEARCH ADVISORY COMMITTEE

The Research Advisory Committee (RAC) was created in 1975 to provide a broadly based co-ordinating and planning group to assist in the Ministry's research activities. The Committee consists of members selected from MOE branches, regions and a medical advisor from the Ministry of Labour. One of the tasks of the RAC is the administration of the Provincial Lottery Funds allotted to the Ministry for health oriented environmental projects.

Research proposals for possible support with MOE Lottery Funds are usually received from outside sources such as universities, private consultants or they may be proposed from within the Ministry for research to be carried out by external agencies. In the fiscal year 1980-1981 for example 25 new proposals were received out of which 6 were funded. Including projects that were continued from previous years, twenty nine projects were funded in the fiscal year for a total budget of approximately 1.8 million dollars. Twelve projects were completed in that year. Table

I shows the main areas of research for which funds have been allocated between 1977 and 1984. The total number of man years spent on these Lottery projects between 1977 and 1981 was 180, consisting of 50 man years of professional and 130 man years of technician and student service.

The Research Advisory Committee held a popular and highly rated one day technology transfer conference in November 1980, during which selected researchers presented their findings to attendees from industry, academia and staff from the Ministry and other government agencies. This year's Conference No. 2 was held in November and information on it can be obtained from:

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Table 1

Division of MOE Lottery Funds Among Selected Important
Categories of Projects

| | Approximate Cost K \$ | % |
|---|--------------------------|-------|
| Clean-up projects including radioactivity in Elliot Lake Lead Contamination and Spills | 1,603.1 | 19.7 |
| Research and Development and other work related to: | | |
| - Acid Rain problems | 53.6 | 0.6 |
| - Pollution from PCBs and other organic chemicals | 703.3 | 8.7 |
| - Drinking water | 451.7 | 5.6 |
| - Sewage Treatment and Disposal | 1,160.5 | 14.3 |
| - Health effects of air pollution and its abatement | 1,541.4 | 19.0 |
| - Solid Waste Disposal | 531.2 | 6.5 |
| - Assessment of substitutes for PCBs as Dielectric Fluids | 222.1 | 2.7 |
| - Miscellaneous Environmental Health Areas | 976.8 | 12.0 |
| - Urban Drainage and Combined Sewers Pollution | 868.9 | 10.9 |
| Total for seven fiscal years | \$8,112.6 | 100.0 |

WATCH OUT FOR BORON!

Little attention has been paid to the element boron as a water pollutant, because its commonly occurring form (borate) is relatively non-toxic. It was not considered a threat to the Great Lakes; indeed the International Joint Commission, in its early years, decided not to monitor for boron. This situation could change. There is a continuing TV advertising campaign promoting detergents containing boron. If successful, one can expect increased levels of boron in the sewers and treatment plant effluents, much of it entering the Great Lakes.

It is of interest that there appears to be an increase in the red algae *Bangia* along the shores of Lake Ontario. It is said to be an invading marine species and it has been suggested that boron may promote its growth.

Recent research in Nova Scotia indicates that under conditions of excess boron, the element can alleviate seasonal nutrient deficiency in some phytoplankton. Filamentous algae are known to be bio-accumulators of a range of metals, as well as boron. Analysis of algae can, therefore, provide useful information where concentrations of boron in the water are below the detection limit for the analytical method.

The Ministry of the Environment's Water Resources Branch is currently conducting a study of metal uptake by various algae; boron is one of the parameters.

Colorimetric methods currently in use (curcumin for water and quinalizarin for vegetation samples) have drawbacks, such as interferences and inadequate sensitivity. The Azomethine-H procedure appeared to be most promising, with application to several types of samples such as waters, sewage and treatment plant effluents, vegetation and

Send solution to:
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RECENT PUBLICATIONS AND PRESENTATIONS

- P. Baulu, O. W. Berg and G. A. V. Rees. A Novel, Automated Liquid Extraction Technique for Aqueous Pesticides Samples. Presented at the Pittsburgh Conference, Atlantic City, March 1981.
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- M. Moselhy and P. Vijan. Simultaneous Determination of Trace Metals in Sewage and Sewage Effluents by Inductively Coupled Argon Plasma Atomic Emission Spectroscopy. Anal. Chem. Acta V130, 1981.
- J. E. Pagel, A. A. Qureshi, D. M. Young and L. T. Vlassoff. Comparison of Four Membrane Filter Methods for Fecal Coliform Enumeration. Submitted to Applied and Environmental Microbiology.
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- G. A. V. Rees, R. D. Smillie, H. Tosine and J. Osborne. Techniques for the Measurement of Chlorinated Dioxins and Related Compounds in Incinerator Effluents. Presented at the 28th Canadian Spectroscopy Society Symposium in Ottawa, September 1981.
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- P. Vijan. Development and Automation of Gas Phase Atomic and Molecular Spectroscopy for Trace Analysis. Presented at the AOAC workshop, Ottawa, May 1981.
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- P. Vijan and G. R. Wood. Automated Determination of Ammonia by Gas Phase Molecular Absorption. Anal. Chem., 53, 1447-1450, 1981.
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